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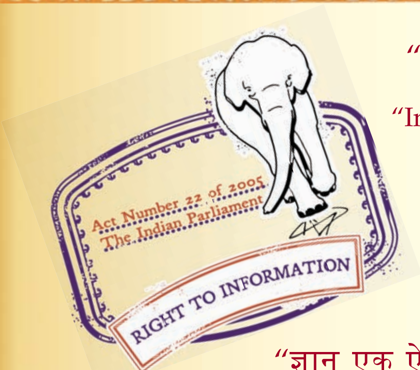
IS 11535 (1986): Method of test for determination of glycosidic hydrocyanic acid in pulses [FAD 16: Foodgrains, Starches and Ready to Eat Foods]



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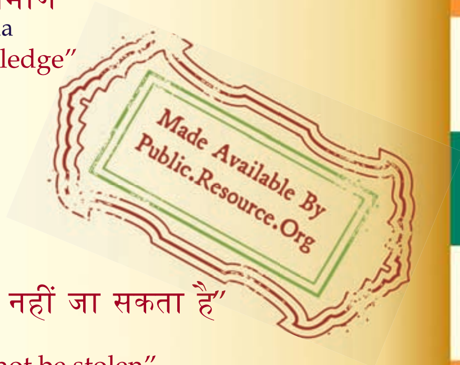

“ज्ञान एक ऐसा खजाना है जो कभी चुराया नहीं जा सकता है”
Bhartṛhari—Nītiśatakam
“Knowledge is such a treasure which cannot be stolen”

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Indian Standard

METHOD OF TEST FOR DETERMINATION OF GLYCOSIDIC HYDROCYANIC ACID IN PULSES

(ISO Title : Pulses — Determination of Glycosidic Hydrocyanic Acid)

Foodgrains and Foodgrain Industries Sectional Committee, AFDC 32 [Ref : Doc : AFDC 32 (2413)]

National Foreword

This Indian Standard, which is identical with ISO 2164 - 1975 'Pulses — Determination of glycosidic hydrocyanic acid', issued by the International Organization for Standardization (ISO), was adopted by the Indian Standards Institution on the recommendation of the Foodgrains and Foodgrain Industries Sectional Committee and approved by the Agricultural and Food Products Division Council.

In the adopted standard, certain terminology and conventions are not identical with those used in Indian Standards, attention is particularly drawn to the following:

a) Comma (,) has been used as decimal marker, while in Indian Standards the current practice is to use full point (.) as decimal marker; and

b) Wherever the words 'International Standard' appear referring to this standard, they should be read as 'Indian Standard'.

The technical committee responsible for the preparation of this standard has reviewed the provisions of the following ISO Standards and has decided that they are acceptable for use in conjunction with this standard :

ISO/R 951 Pulses — Sampling

ISO 2170 Cereals and pulses — Sampling of milled products

For determining compliance with the test requirements prescribed in the standard, result of a test or analysis shall be rounded off in accordance with IS : 2 - 1960 'Rules for rounding off numerical values (*revised*)'.

Adopted 27 January 1986	© July 1986, ISI	Gr 3

INDIAN STANDARDS INSTITUTION
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

1 SCOPE AND FIELD OF APPLICATION

This International Standard specifies a method for the determination of glycosidic hydrocyanic acid in pulses.

The method is generally applicable but may require modification if sulphides or certain other sulphur compounds are present. Conversely, if no such compounds are present, a mercurimetric titration procedure may be used, details of which are given in the annex.

2 REFERENCES

ISO/R 951, *Pulses – Sampling.*

ISO 2170, *Cereals and pulses – Sampling of milled products.*

3 DEFINITION

glycosidic hydrocyanic acid in pulses : Hydrocyanic acid, determined according to the method described.

The content of glycosidic hydrocyanic acid is expressed as milligrams of hydrocyanic acid (HCN) per kilogram of product.

4 PRINCIPLE

After hydrolysis of the glycosides, steam distillation of the hydrocyanic acid liberated.

Determination of the quantity of acid obtained :

– either by direct titration of the acid in the distillate with silver nitrate solution in an ammoniacal medium and in the presence of potassium iodide, the hydrocyanic acid forming the soluble complex $Ag(CN)_2^-$ and the end point of the titration being characterized by the appearance of permanent turbidity due to precipitation of silver iodide;

– or by back titration of an excess of silver nitrate solution with ammonium thiocyanate solution, in a sufficiently dilute nitric acid medium and in the presence of iron(III) ions, the hydrocyanic acid being precipitated as silver cyanide (AgCN).

5 REAGENTS

All reagents shall be of analytical quality; the water used shall be distilled water or water of at least equivalent purity.

5.1 Potassium dihydrogen phosphate (KH_2PO_4), 20 g/l solution.

5.2 Sodium acetate, 20 g/l solution brought to pH 5,0 ± 0,5 with a few millilitres of acetic acid.

5.3 Sodium hydroxide, in pellets.

5.4 Ammonia solution, about 6 N (ρ_{20} 0,96 g/ml), obtained by diluting concentrated ammonia solution, ρ_{20} 0,92 g/ml, with an equal volume of water.

For direct titration .

5.5 Silver nitrate, 0,01 N standard volumetric solution.

5.6 Potassium iodide (KI), 50 g/l solution.

For back titration :

5.7 Nitric acid, ρ_{20} 1,38 g/ml.

5.8 Silver nitrate, 0,02 N standard volumetric solution.

5.9 Ammonium thiocyanate, 0,02 N standard volumetric solution.

5.10 Indicator, prepared by mixing one part by volume of the nitric acid (5.7) and one part by volume of saturated ammonium iron(III) sulphate solution.

6 APPARATUS

Usual laboratory apparatus not otherwise specified, and the following :

6.1 Mechanical grinding mill, easy to clean, enabling samples to be ground without becoming heated and without appreciable change in moisture content.

6.2 Sieves, with 1 mm apertures.

6.3 Balance.

6.4 Incubator, adjusted to operate at 38 ± 2 °C.

6.5 Steam distillation apparatus, provided with a 1 l flask with ground neck. It should be possible to stopper this removable flask hermetically with a ground stopper. The end of the condenser shall be provided with an extension drawn out to a point.

6.6 Ice-water bath.

For direct titration :

6.7 Volumetric flask, 250 ml.

6.8 Pipette, 100 ml.

For back titration :

6.9 Volumetric flasks, 250 ml and 500 ml.

7 SAMPLING

Prepare a final lot sample of appropriate size in accordance with ISO/R 951 or ISO 2170.

8 PROCEDURE

8.1 Preparation of test sample

Grind about one-twentieth of the laboratory sample in the previously well-cleaned mechanical grinding mill (6.1), in order to complete the cleaning of the mill, and reject these grindings. Then grind the rest to particles which will pass through the sieve (6.2) completely, collect the grindings, mix thoroughly and carry out the determination without delay.

In the case of ground pulses, use the final lot sample.

8.2 Test portion

Weigh, to the nearest 0,1 g, approximately 20 g of the test sample (8.1).

8.3 Determination

8.3.1 *Methods of hydrolysis*

Either of the two alternative procedures which follow (8.3.1.1 or 8.3.1.2) may be used.

If the enzymic power of the sample seems insufficient, enzymic hydrolysis should be the preferred method.

8.3.1.1 ENZYMIC HYDROLYSIS

Transfer the test portion (8.2) to the 1 l flask (6.5). Add 50 ml of water containing 2 crushed sweet almonds (total mass 1,5 to 2,0 g). Stopper the flask hermetically and leave it for 2 h in the incubator (6.4) at 38 °C.

NOTE — Verify that the almonds used do not contain any hydrocyanic acid that can be liberated under the conditions of the test.

Cool the flask and its contents in the ice-water bath (6.6).

Add 80 ml of water, 10 ml of the sodium acetate solution (5.2) and a drop of anti-foaming agent. Fit the flask immediately to the distillation apparatus (6.5).

8.3.1.2 ALTERNATIVE HYDROLYSIS PROCEDURE

Transfer the test portion (8.2) to the 1 l flask (6.5) and add 50 ml of distilled water and 10 ml of the sodium acetate solution (5.2) or potassium dihydrogen phosphate solution (5.1). Stopper hermetically, mix well and leave the flask for 12 h in the incubator (6.4) at 38 °C.

Cool the flask and its contents by immersion in the ice-water bath (6.6) and add 80 ml of water and a drop of anti-foaming agent; fit the flask to the distillation apparatus (6.5) immediately.

8.3.2 *Distillation*

Distil, collecting approximately 100 ml of distillate.

If the direct method of titration (8.3.3.1) in the presence of potassium iodide (5.6) is to be used, collect the distillate in 20 ml of distilled water containing 1 g of sodium hydroxide (5.3). (Solution A.)

If the back titration method (8.3.3.2) with the ammonium thiocyanate solution (5.9) is to be used, collect the distillate in a volume (20 to 50 ml) of the 0,02 N silver nitrate solution (5.8), to which has been added 1 ml of the nitric acid (5.7). (Solution B.) The volume of silver nitrate solution used depends on the presumed content of hydrocyanic acid.

8.3.3 *Titration*

8.3.3.1 DIRECT TITRATION

Transfer solution A to a 250 ml volumetric flask (6.7), dilute to the mark with distilled water and mix thoroughly. Pipette 100 ml into a beaker. Add 2 ml of the potassium iodide solution (5.6) and 1 ml of the ammonia solution (5.4).

Titrate with the 0,01 N silver nitrate solution (5.5) until permanent turbidity appears. For the easy recognition of the end point of the titration, it is recommended that a black background be used.

Make a second titration with another 100 ml portion of distillate and take the mean of the two titrations.

8.3.3.2 BACK TITRATION

Transfer solution B to a 500 ml volumetric flask (6.9) and dilute to the mark with water. Mix thoroughly and filter through a dry filter, collecting the filtrate in a dry vessel.

Add 2 to 3 ml of the indicator (5.10) to 250 ml of filtrate (measured in a volumetric flask (6.9)), and titrate with the ammonium thiocyanate solution (5.9), while shaking, until a permanent brown-red coloration appears.

8.3.4 Carry out two determinations on the same test sample.

8.4 Test for sulphides

A nearly black precipitate denotes the presence of a significant quantity of sulphides, whereas a brownish precipitate denotes only a very slight sulphide content (see clause 10).

8.5 Blank test

Carry out a blank test under the same conditions as in the determination proper but replacing the distillate by distilled water.

9 EXPRESSION OF RESULTS

9.1 Direct titration

The content of glycosidic hydrocyanic acid, expressed in milligrams of hydrocyanic acid (HCN) per kilogram of sample, is equal to

$$0{,}54\,(V_0 - V_1) \times \frac{250}{100} \times \frac{1\,000}{m} = \frac{1\,350\,(V_0 - V_1)}{m}$$

where

m is the mass, in grams, of the test portion:

V_0 is the volume, in millilitres, of the silver nitrate solution (5.5) used for the determination proper;

V_1 is the volume, in millilitres, of the silver nitrate solution (5.5) used for the blank test.

9.2 Back titration

The content of glycosidic hydrocyanic acid, expressed in milligrams of hydrocyanic acid (HCN) per kilogram of sample, is equal to

$$0{,}54\,(V_3 - V_2) \times \frac{500}{250} \times \frac{1\,000}{m} = \frac{1\,080\,(V_3 - V_2)}{m}$$

where

m is the mass, in grams, of the test portion;

V_2 is the volume, in millilitres, of the ammonium thiocyanate solution (5.9) used for the determination proper;

V_3 is the volume, in millilitres, of the ammonium thiocyanate solution (5.9) used for the blank test.

NOTE (to 9.1 and 9.2) — If the standard volumetric solutions used are not of exactly the strength indicated in the list of reagents, a suitable correction factor should be used in calculating the results.

9.3 Take as the result the arithmetic mean of the two determinations.

Express the result to one decimal place.

NOTE — If the result obtained is less than 10 mg of hydrocyanic acid per kilogram of sample, consider the sample practically free from glycosidic hydrocyanic acid.

10 NOTE ON PROCEDURE

If the distillate contains sulphides or sulphur compounds, a more or less abundant black precipitate appears through action on silver ions. In this case it is advisable to repeat the procedure using only the direct titration method (8.3.3.1), modified by replacing the first paragraph by the following :

Transfer the distillate to a 250 ml volumetric flask (6.7), dilute to the mark and mix thoroughly. Pipette 100 ml into a beaker. Add 5 ml of lead nitrate solution (5 g/l) and mix. After allowing to stand for 15 min, filter and wash the beaker, the precipitate and the filter three times, using 10 ml of water each time. Add to the filtrate and washings 2 ml of the potassium iodide solution (5.6) and 1 ml of the ammonia solution (5.4).

11 TEST REPORT

The test report shall show the method used, the mode of hydrolysis and the mode of titration followed and the result obtained. It shall also mention any operating conditions not specified in this International Standard, or regarded as optional, as well as any circumstances that may have influenced the result.

The report shall include all details for complete identification of the sample.

ANNEX

DETERMINATION OF GLYCOSIDIC HYDROCYANIC ACID BY THE MERCURIMETRIC METHOD

A.0 INTRODUCTION

If the distillate does not contain sulphides or sulphur compounds, the content of glycosidic hydrocyanic acid in pulses can also be determined by back titration with a solution of mercury(II) nitrate.

For this purpose it is convenient to apply the method described in this International Standard, with the following modifications to the clauses and sub-clauses mentioned below.

A.1 PRINCIPLE

Modification of the back-titration method by replacement of the silver nitrate solution by mercury(II) nitrate solution.

A.2 REAGENTS

Replace reagent 5.8, "**Silver nitrate**, 0,02 N standard volumetric solution", by "**Mercury(II) nitrate**, 0,02 N standard volumetric solution".

A.3 PROCEDURE

A.3.1 Distillation

Replace the last two paragraphs of 8.3.2 by :

"Collect the distillate in a volume (20 to 50 ml) of 0,02 N mercury(II) nitrate solution (A.2), to which has been added 1 ml of nitric acid (5.7). (Solution B.) The volume of mercury(II) nitrate solution used depends on the presumed content of hydrocyanic acid."

A.3.2 Back titration

Replace 8.3.3.2 by :

"Add 1 to 2 ml of the indicator (5.10) to solution B, cool to a temperature below 15 °C and titrate with the ammonium thiocyanate solution (5.9), while shaking, until a permanent brown-red coloration appears.

Maintain the temperature below 15 °C until titration is complete."

A.4 EXPRESSION OF RESULTS

Back titration

Replace the first paragraph of 9.2 by :

"The content of glycosidic hydrocyanic acid, expressed in milligrams of hydrocyanic acid (HCN) per kilogram of sample, is equal to

$$0{,}54\,(V_3 - V_2) \times \frac{1\,000}{m} = \frac{540\,(V_3 - V_2)}{m}\text{ "}$$

Reprography Unit, ISI, New Delhi